PROJECT TITLE

QA ANALYTICAL SERVICES

PERIOD COVERED

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WRITTEN BY

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#### 1.5. LEGISLATION

- Denaturation of rum and ethanol intended for UK cigarettes.

The question was raised by PMG-Munich as to which denaturation agents are allowed to be used in the UK (1).

The problem was examined by "R+D Technical Services" (2):

- According to list 3 of the Hunter list:
  Ethanol and rum, which may be used as additives in tobacco products, must be denaturated with not more than
  0.05 % of nicotine salts (calculated as nicotine).
- According to list 2 of the Hunter list:
  Butane-2-one (methyl ethyl cetone) is mentioned in the list of products which may be applied to the tobacco blend. Their total level must not exceed 0.15 % of the tobacco blend in cigarettes, calculated on a dry weight basis.

Butane-2-one and ethanol have the same boiling point and are evaporated together during tobacco processing. The residual amount of the former will not significantly increase the total amount of additives put in the blend which are mentioned in list 2. As Butane-2-one is already used as a denaturation agent for ethanol in Germany, we recommend its use for the preparation of AC-solutions for UK cigarettes (3).

# 2.1. CASING KITCHEN

- MAG-AC

The preparation procedure was established for this new solution (4).

- PZ-8-FI

The preparation procedure had to be modified following the acquisition of a new recipient (5):

## 3.2.1. TRIALS WITH TOBACCO INGREDUENTS

- Invert sugar (M. BORGWALDT, Hamburg; "GLYCARMEL 4952" / yellow sheet 6189, 100 kg)

This product is used as a standard material in PMG. An industrial trial (MDF-CH) was organized in order to qualify this product for FTR.

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Laboratory: The sample is within specifications. Smoke analyses: No significant difference between the standard and the trial was found.

Subjective evaluation (Panel B, discriminative test):
A significant difference (99 to 99.5 %) between the trial and the standard was found.

Furthermore, as the drum did not have the usual dimensionable product could not be liquified on the installation in the casing kitchen.

After discussions with the supplier concerning the dimensions of the drums, the trial might be repeated.

#### - Liquid sugar

Liquid sugar instead of raw cane sugar is used by PMH for the preparation of BURLEY CASING. For handling and price reasons, the possibility of applying liquid sugar in PMG-Berlin and Munich was studied.

Four reference samples of different shipments to PMH were analysed:

|   | Fructose+Glucose<br>(HPLC) (%) | Saccharose (HPLC) (%) | Water (KF) |
|---|--------------------------------|-----------------------|------------|
| 1 | 10.5                           | 56.8                  | 33.3       |
| 2 | 10.3                           | 50.9                  | 36.8       |
| 3 | 8.8                            | 56.9                  | 32.5       |
| 4 | 22.4                           | 43.7                  | 33.1       |

Furthermore the influence of the storage time was studie ) on a fresh sample:

|                  | Fructose+Glucose |      | Saccharose |     |
|------------------|------------------|------|------------|-----|
|                  | (HPLC)           | (%)  | (HPLC)     | (용) |
| Upon delivery    | 1                | 12.8 |            |     |
| After two months | 1                | .9.5 | 50.2       |     |

The above-mentioned results show a certain doubt about the regularity of the shipments as regards the composition of different sugars. Furthermore, depending on the storage time and conditions, a decomposition of saccharose into fructose and glucose seems to take place.

For this reason, and also because commercially-available samples from different suppliers show considerable differences in the composition of the sugars, their influence on the cigarette taste was evaluated.

Test-cigarettes (MLF-IT) were produced in PMG-Munich with the following test materials:

|   | **************************************   | Fructose + Glucose (HPLC) (%) | Saccharose (HPLC) (%) | Water (KF):<br>(%) |
|---|--|-------------------------------|-----------------------|--------------------|
| - | Invert sugar<br>SUCRERIE TIRLEMONT(BE)   | 77.6                          | < 0.5                 | 20.2               |
| - | Liquid sugar<br>SÜDZUCKER (GER)          | -<br>2.3 *                    | 65.6<br>64.2 *        | 33.2               |
| - | Liquid sugar<br>COMMERCIALE SUCRERIE(FR) | 22.8 *                        | 65.9<br>44.5 *        | 32.9               |
| - | Liquid sugar<br>PFEIFFER&LANGEN (GER)    | 41.4<br>45 *                  | 27.5<br>23 *          | 31.1               |

\* indication of the supplier

The total amount of sugar was identical in all the trials; it was calculated on the basis of the standard recipe of BURLEY CASING.

Smoke and tobacco analyses: No significant difference between the standard and the trials was found except in the contents of reducing sugars which reflect the quantity of the additions.

Subjective evaluation (Panel A): A significant difference between the standard and the trials was found. None of the trials were accepted (6).

A further trial (MLK-21) with the standard material used in PMH (supplier: SUIKER UNIE) was carried out by PMG-Berlin.

Laboratory: The composition of the sample was found to be as follows:

| Fructose+Glucose (HPLC) | (ક) | 12.4 |
|-------------------------|-----|------|
| Saccharose (HPLC)       | (%) | 54.4 |
| Water (KF)              | (%) | 33.0 |

Smoke and tobacco analyses: No significant difference between the standard and the trial was found except in the contents of reducing sugars.

Subjective evaluation (Panel A): A significant difference between the standard and the trial was found. The trial was refused (7).

A further repetition of this trial in FTR, including a panel D test, is planned.

# 3.2.2. QUALITY CONTROL OF TOBACCO INGREDIENTS

- Honey (J. SCHUETTE & CO, Bremen (GER) / blue sheet 9084)

Two drums out of 33 had to be refused. Reason: In one drum an accumulation of solid particles (impurities) was found on the surface. The contents of the other drums had a strongly pronounced acid taste. This was probably brought about by the fermentation which was then stopped by heat treatment.

## 3.4.1. TRIALS ON FILTER COMPONENTS

- Activated charcoal (CHEMVIRON, Zürich; "UKCT"/yellow sheet 5418, 14.4 t and "MF III" / yellow sheet 5428, 14.4 t)

These materials were tested on their own or blended with the standard charcoal from PICA. The results were negative (8) The Purchasing Department proposed blending the above-mentioned quality with charcoal from PICA. In this case the former made up 5 % of the blend. This operation would take place in the production center in Levallois (FR) (9). However, the installation in Onnens, producing the mixture "CAPO 46", has a free capacity. Blending one quality of CHEMVIRON charcoal in a 1:10 ratio to PICA charcoal would occupy the installation for 93 days (10).

It was decided to run a trial with 10 % charcoal from CHEMVIRON blended with 90 % charcoal from PICA. Reason: The installation in Onnens guarantees a homogeneous blend and the operations would be supervised by QA.

## "UKCT":

Laboratory: The results of the blend are within specification. Smoke analyses: No significant difference between the trial and the standard was found. Subjective evaluation:

- Panel B (discriminative test): No significant difference between the trial and the standard was found.
- Panel D: 193 answers were taken into consideration for statistical evaluation. No significant difference was stated between the trial and the standard (11).
- Panel A: A significant difference between the trial and the standard was found. The trial was refused.

## "MF III":

Laboratory: The results of the blend are within specifications.

Smoke analyses: No significant difference between the trial and the standard was found. Subjective evaluation:

- Panel B (discriminative test): No significant difference between the trial and the standard was found.
- Panel D : 216 answers were taken into consideration for statistical evaluation. No significant difference was stated between the trial and the standard (12).
- Panel A: A significant difference between the trial and the standard was found. The trial was refused.
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# 3.7.1. TRIALS WITH CIGARETTE SEAM GLUES

- "SICHOCOLL STN 5033" (HENKEL AG, Düsseldorf (GER))

This type is a starch-based paste. PMG-Munich evaluated this quality on MLF-IT cigarettes with a view to finding an alternative to its own production.

Machineability: Better than the standard glue. Smoke analyses: No significant difference between the standard and the trial was found. Subjective evaluation: (Panel A): No significant difference between the standard and the trial was found. The trial was accepted.

On the basis of these results it was decided that this type can be used as a standard material (13).

## 3.7.2. QUALITY CONTROL OF CIGARETTE SEAM GLUES

- "LESSO 1487 X-3" (LAESSER AG, Erlinsbach)

This is the standard starch-based liquid glue which is applied through a KAYMICH-nozzle. The producer guarantees the quality of this liquid glue for three months as from its day of production.

In the beginning of December, simultaneously in the production centers of Serrières and Cousset, the glue was found to be too liquid. Even by applying it at room temperature and without pressure, serious glueing problems were encountered. An inspection of all the drums stored in Serrières, Cousset and Onnens showed that part of the glue had started to ferment. Fermentation causes a rapid decrease in pH and viscosity. One of the most affected drums showed for example a pH of 5.4 instead of > 7.4 and a viscosity of 1600 cps instead of > 14900 cps.

All the affected drums were blocked immediately and replaced by new ones. During the period of December 81 and January 82 a beginning of fermentation was detected during the reinforced control on further shipments of this glue produced at different dates. 25 out of 65 drums had to be refused.

Up till now, the producer has not been able to give any explanation of this decrease in quality.

The following measures were taken:

- The glue is produced every Friday and supplied every Tuesday. The quantity ordered by FTR just covers our needs from one supply-day to the next. This means that the glue has been entirely used up 12 days after its production.
- The changes in the glue application systems planned for Serrières and PMG-Munich have been suspended until the quality problems of the glue have been resolved.

# 3.14.2. QUALITY CONTROL OF ETNA

- A new manual for Quality Control was established (14).

  The following main modifications were introduced into the new manual (15):
  - All analyses on the cut rag are done by the Production Control in Serrières.
  - Chemical analyses are only done on expanded tobacco.
     A reference sample of cut rag will be retained.
  - All physical analyses on the expanded tobacco after storage have been abandoned, except for the determination of the filling power.

# 3.15.2. QUALITY CONTROL OF RCB

- A new manual for Quality Control was established (16).

  The following main modification was introduced in this new edition (17):
  - Weekly chemical analyses are done on the final product. However, regular TL-analyses will only be done every four months, instead of every month.

# 3.16.2. QUALITY CONTROL OF ESTHER

- A manual for Quality Control was established (18).

  The following main modification, compared with the routine procedure, was introduced:
  - Chemical analyses on the final product are performed on every fifth batch per blend.

# 5.3. ASSISTANCE TO OTHER AFFILIATES

- Analyses for the ETNA-plant in PMH-Bergen op Zoom Humectants in tobacco: (18 samples)
- Analyses for the ETNA-plant in PMG-Munich Humectants in tobacco (2 samples)

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- l Letter from Janke-W to Lopes-F (January 11 1982)
- 2 Letter from Keagy-K.B. to Widmer-A (February 5 1982)
- 3 Letter from Widmer-A to Janke-W (February 15 1982)
- 4 Letter from Schwarb-A to the CASING KITCHEN (February 3 1982)
- 5 Letter from Schwarb-A to the CASING KITCHEN (February 8 1982)
- 6 Letter from Widmer-A to Lutzig-B.W. (March 20 1981):
- 7 Letter from Widmer-A to Tessendorf-W (February 15 1982)
- 8 Monthly report Widmer-A (February 1981)
- 9: MPP & QA Coordination Meeting (April 4 1981)
- 10 Letter from Caccivio-J.P. to Schwarb-A (October 14 1981)
- 11 Letter from Stampfli-M to Widmer-A (December 1 1981)
- 12 Letter from Stampfli-M to Widmer-A (February 4 1982)
- 13 Letter from Widmer-A to Lutzig-B.W. (February 15 1982)
- 14 "ETNA/procédé contrôle qualité" from Widmer-A and Matthey-A (February 15 1982)
- 15 "ETNA/procédé contrôle qualité" from Widmer-A (January 8 1981)
- 17 "Cahier des charges, MONIQUE" from Joseph-L (May 1979)
- 18 "ESTHER / procédé contrôle qualité" from Widmer-A and Matthey-A (February 15 1982)

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